

## PATENT ABSTRACTS OF JAPAN

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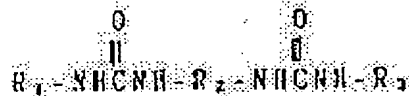
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## (54) UREA GREASE COMPOSITION

(57)Abstract:

PURPOSE: To provide a grease compsn. with a long life at high temp. and excellent in noise reduction by compounding a specified amt. of a specified diurea compd. as a thickening agent in a base oil wherein an alkyldiphenyl ether is an essential component.

CONSTITUTION: A base oil is prepd. by compounding an alkyldiphenyl ether obtd. by the addition reaction of diphenyl ether with a 10-22C  $\alpha$ -olefin as an essential component and, if necessary, a mineral oil and an ester type synthetic oil. A grease compsn. is prepd. by compounding 2-25wt.% diurea compd. of the formula (wherein R2 is a 6-15C arom. hydrocarbon group; R1 and R3 are each 8-18C linear alkyl and the content of 8C alkyl in R1 and R3 is 60-100mol%) as a thickening agent in this base oil. The compd. of the formula is obtd. by reacting a diisocyanate (e.g. 2,4-tolylene diisocyanate) with a monoamine (e.g. n-octylamine).



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(57)

[WHAT IS CLAIMED IS:]

[Claim 1]

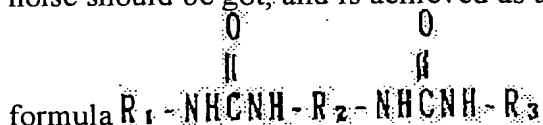
In; In the base oil which is essentially based on alkyl diphenyl ether oil, as thickener, next nitramine



(aromatic system hydrocarbon group of carbon number 6-15,  $\text{R}_1$  extend to  $\text{R}_2$  in equation, and  $\text{R}_3$  is carbon number 8 and 18 normal chain alkyl group;) Grease composition; wherein; With diurea chemical agent 2 expressed in) which was  $\text{R}_1$  and rate 60 of said alkyl group of carbon number 8 to occupy within  $\text{R}_3$  - 100 mol % - 25 % by weight was combined.

## [DETAILED DESCRIPTION OF THE INVENTION]

(a field of industrial application) it is lightweighted and progress of machine art of late years is remarkable, and, miniaturization of machine part, speedup of bearing by promotion of efficiency advances high, a lubrication point tends to become high temperature. In addition, On the other hand, noise in bearing revolution becomes issue. The present invention relates to urea grease composition superior in the low noise character which longlivedness is provided under such a high temperature, and win. (prior art:), under such a high, urea grease using synthetic oil is employed by base oil. By way of example only, In Japanese Patent Application No. 63-86631 by a this application person, longlivedness is enabled under high temperature in urea grease with the use of alkyl diphenyl ether oil in synthetic oil. It is the lubricant that compound using powder of polytetrafluoroethylene was good in heat resistance in thickener, and, besides urea grease, Japanese Patent Application No. 63-164868 by a this application person are given. (only a problem to be solved by the invention) while doing, as for the urea grease of these complex base oil and the compound, is not always satisfied with the only low noise which is superior in heat resistance. This low noise is done with essential condition in electric motor of auto called for a good point of ride comfort as well as electric motor of home electric appliance. Therefore, It becomes a problem to provide grease longlivedness is comprised under high to answer these claim and is superior in noise low. (a means for solving problem) in the base oil which is essentially based on alkyl diphenyl ether oil with the thing which grease the present invention comprises longlivedness under high temperature and combine with low noise should be got, and is achieved as a result of analysis zealously, as thickener, next general



(aromatic hydrocarbon group of carbon number 6-15,  $\text{R}_1$  and  $\text{R}_3$  is normal chain alkyl group of carbon number 8 and 18, and equation medius  $\text{R}_2$  is  $\text{R}_1$  and rate 60 of the alkyl group of carbon number 8 occupying within  $\text{R}_3$  - 100 mol %) grease composition including having combined with expressed diurea compound 2 - 25 % by weight is related to. Alkyl diphenyl ether oil used for the present invention is provided by addition reaction of 1-3 alpha - olefin mol of one diphenyl ether mol and carbon number 10-22, attribute is different by means of carbon number of alpha - olefin, application mol number, but, it is yellow transparence liquid from achromatism, and it is it in itself publicly known substance. As for alkyl diphenyl ether oil, chain business smooth oil employed under high temperature by diffusion pump oil, Japanese Patent Publication No. 58-22515 bulletin is disclosed at the rotary pump oil that essential constituent does alkyl diphenyl ether oil at Japanese Patent Publication No. 51-44263 bulletin, Japanese Patent Publication No. 52-1722 bulletin by sea water sexual hydraulic oil resistance, Japanese Patent Publication No. 52-24628 bulletin. The present

invention is synthesis hydrocarbon oil represented in ester synthetic oil represented in, by way of example only, the base oil which it is made contain in to get in grease, preferably base oil 50 % by weight, it is necessary, it can be used together liquid petrolatum and diester, tetraester and poly - alpha - olefin with but it is not limited particularly content of alkyl diphenyl ether oil high speed spin condition longlivedness. In addition, Diurea compound expressed in equation used as thickener (1) is conventional, and, with a thing provided in reaction of diisocyanate and monoamine, formula (1) medius  $R_1$  and  $R_3$  is normal chain alkyl group of carbon number 8 and 18 in the present invention, and it is  $R_1$  and rate 60 of the alkyl group of carbon number 8 occupying within  $R_3$  - 100 mol %, but, with under mol %, comparing unfavorably, leak from bearing are massive in shear stability and heat resistance, and reason 60 as thus described to do is because application of long time is not born. For diisocyanate becoming  $R_2$  after the reaction, it is 2,4- tolylene diisocyanate, 2,6 tolylene diisocyanate, 4,4 diphenyl-methane -  $\square$ OE - diisocyanate, 1,5- *nafuchiren* diisocyanate and these mixture, and straight-chain amine of  $R_1$ ,  $R_3$  and carbon number 8 and 18 is n- octyl amine and n- octadecylamine. Blending quantity of the diurea compound is 2 - 25 % by weight as against the grease, but, it is not suitable as grease lubricating bearing both so that individualization assumes grease provided with under double quantity % is floating and that 25 % by weight is exceeded. In addition, In grease composition of the present invention, additive such as oxidation inhibitor, anti-rust agent, extreme pressure agent, abrasion proof agent, oiliness agent, individual lubricant can be doped if necessary. (action) The alkyl diphenyl ether oil which employed to the present invention comprises enough viscosity under high temperature, there is a little generation of sludge, too there is a little vaporization under high temperature and and it is more superior in oxidation stability than liquid petrolatum and conventional synthetic oil namely ester oil and poly - alpha - olefin oil. Because of this, These mineral oil and conventional synthetic oil are compared with base oil and the grease which did, and grease composition with the present invention comprises longlivedness under high temperature. However, Merely, in base oil, using alkyl diphenyl ether oil, hold, and then this long-life ability is not provided, the combination that is partial response with thickener becomes important. By way of example only, As for the case using the *yukika* bentonite which is representative thickener of heat-resistant mold, this longlivedness ability is not provided. In addition, The grease which assumed urea compound and polytetrafluoroethylene depending on the present invention thickener comprises longlivedness under high temperature, but, the low noise which is another requisite ability is not satisfied. It is known that this, noise evolution depends upon class of thickener of grease greatly, but, the grease which assumed urea chemical agent with the present invention thickener is superior in noise low. A reason to be good in this low noise does not have positive evidence, but, it is thought that thickener particle morphology is depended upon. Grease holds oil in capillary force within the three-dimensional anastomosis which this thickener grain composes, semisolid is had a configuration of, and it holds. These thickener grain is crystalline, and various morphology is caught by class of thickener. The configuration is dilute as shown in electron microgram of figure 1 (magnification 6000 times), and, by way of example only, thickener grain of the grease with the present invention, configuration of thickener corpuscule takes crystal morphology such as for example tabular corpuscule to have a short as shown in electron microgram of figure 2 (magnification 6000 times) thick or this aggregate as a thing of the aromatic subject end of urea radical thickener of grease as shown in Japanese Patent Application No: 63-86631 specification for what take slim tenioid crystal morphology. When grease is employed by bearing, these thickener grain intervenes in lubrication department, too, but, as for the dilute corpuscule, as for the thick crystal grain, is hard to be broken for what is easy to be cut easy, oscillation is given bearing. It is this jolt namely noise, and it gets together, and it is thought that grease of thickener composition with the present invention is superior in noise low. Satisfaction occupied the grease composition with the present invention comprised longlivedness under high temperature than the above and that it was superior in low noise characteristic. It is explained in embodiment concretely in the following. (embodiment) the present invention is explained by embodiment and a comparative example concretely as follows. In addition, Table 1 and blending component shown in 2 are based on, and, in each embodiment and a comparative example, grease is produced, by method shown in next, physical property and ability were evaluated. (a) Misce butterfly degree ... JIS K 2220 is

depended on. (b) It was examined by bearing sound check ... *anderonmeta*. Check condition: Quantity of bearing 608, rounds per minute equation 1 800rpm thrust load 2kg grease charge 0.35ml appraisal: It is shown with a mark (one hundred perfect score) than the number of the noise, result of *anderonreberu*. (c) Bearing lubricity life test ... ASTM D 1741 is depended on. But, The check temperature assumed 150 degrees Celsius. In example 1 to 4 and 1-8 comparative example reaction vessel, moiety and monoamine tota massa of base oil shown in table 1 are taken, it was warmed to 70-80 degrees Celsius. In the other container, base oil moiety and diisocyanate tota massa are taken, and it is warmed to 70-80 degrees Celsius, this was stirred as well as reaction time. For exoergic reaction, temperature of reactant rises, but, in this state, a churn is continued for about 30 minutes, after having done reaction enough, it rises, it held with 170-180 for 30 minutes, and it was cooled. This is kneaded in three steps of roll mill, it was assumed objective grease. In alkyl diphenyl ether oil tota massa shown in comparative example table 9 3, *yukika* event tota massa is taken, after having stirred in room temperature well, acetone 30.0g are added, heat / stirring is continued by 60 degrees Celsius, it was cooled. This is kneaded by three steps of roll kill, it was assumed objective grease. In *arukirujifunirueteru* oil tota massa shown in comparative example table 10 3, polytetrafluoroethylene tota massa is taken, it was stirred in room temperature well. This is kneaded in three steps of roll mill, it was assumed objective grease. In addition, As for the embodiment and the alkyl diphenyl ether oil shown in a comparative example, kinematic viscosity of 40 degrees Celsius employed 97.0cSt, viscosity index 124, a thing having attribute of fire point 286 degrees Celsius. In addition, List of pen Tae re-torr ester oil, poly - alpha - olefin oil, fire point employed a thing having attribute of 254 degrees Celsius, 285 degrees Celsius 29.6cSt, 46.0cSt, viscosity index 124, 135 each, kinematic viscosity of 40 degrees Celsius.

表 - 1 実施例 1 ~ 4

		実施例 1	実施例 2	実施例 3	実施例 4
基油	(g)	アルキルジフェニル エーテル油 1760.0	アルキルジフェニル エーテル油 1790.0	アルキルジフェニルエ ーテル油 880.0	アルキルジフェニル エーテル油 895.0
		—	—	ポリ- $\alpha$ -オレフィン 油 880.0	ポリ- $\alpha$ -オレフィ ン油 895.0
ジイソシアネート (g)		ジフェニルメタン- 4,4'-ジイソシアネ ート 118.1	ジフェニルメタン- 4,4'-ジイソシアネ ート 84.7	ジフェニルメタン- 4,4'-ジイソシアネ ート 118.1	ジフェニルメタン 4,4'-ジイソシアネ ート 88.6
モノア ミン (g)	n-オクタール アミン	121.9	52.4	121.9	64.1
	n-オクタデ シルアミン	—	72.9	—	57.3
(1)式中のR <sub>1</sub> およびR <sub>2</sub> 中 に占める炭素数8のアル キル基の割合(モル%)		100	60	100	70
グリースに対するジウ レア化合物の配合量 (重量%)		12.0	10.5	12.0	10.5
混和ちょう度 25℃		260	261	255	250

	実施例 1	実施例 2	実施例 3	実施例 4
軸受音響 (点)	79	84	78	82
軸受潤滑寿命 (h)	2020	1340	1580	1200

表 - 2 比較例 1 ~ 8

	比較例 1	比較例 2	比較例 3	比較例 4	比較例 5	比較例 6	比較例 7	比較例 8
基油 (g)	鉱油 1760.0	ペンタエリスリトール エステル油 1700.0	ポリ- $\alpha$ - オレフィン 油 1760.0	アルキルジ フェニルエ ーテル油 1760.0	アルキルジ フェニルエ ーテル油 1520.0	アルキルジ フェニルエ ーテル油 1720.0	アルキルジ フェニルエ ーテル油 1964.0	アルキル フェニル ーテル油 1480.0
ジイソシ アネート (g)	ジフェニル メタン- 4,4'-ジイ ソシアネート 118.1	ジフェニル メタン- 4,4'-ジイ ソシアネート 147.6	ジフェニル メタン- 4,4'-ジイ ソシアネート 118.1	ジフェニル メタン- 4,4'-ジイ ソシアネート 85.2	ジフェニル メタン- 4,4'-ジイ ソシアネート 275.2	ジフェニル メタン- 4,4'-ジイ ソシアネート 156.3	ジフェニル メタン- 4,4'-ジイ ソシアネート 17.7	ジフェニ メタン- 4,4'-ジイ ソシアネ ート 255.1
モノアミン (g)	n-オクチ ルアミン 121.9	n-オクチ ルアミン 152.4	n-オクチ ルアミン 121.9	n-オクチ ルアミン 26.4	アニリン 204.8	シクロヘキ シアミン 123.7	n-オクチ ルアミン 18.3	n-オクチ ルアミン 264.1
	—	—	—	n-オクタ デシルアミ ン 128.4	—	—	—	—
(1)式中の R <sub>1</sub> および R <sub>2</sub> 中に占 める炭素 数8のアル キル基の 割合 (モル%)	100	100	100	30	0	0	—	—
グリース に対する ジウレア 化合物の 配合量 (重量%)	12.0	15.0	12.0	12.0	24.0	14.0	1.8	26.0
混和ち ょう度25℃	265	272	251	258	260	252	440< (流動状)	固体状に き製造不
軸受音響 (点)	77	78	68	84	28	22	試験不能	—
軸受潤滑 寿命 (h)	320	240	800	280	2000	1600	試験不能	—

表-3 比較例 9・10

	比較例 9	比較例 10
基油 (g)	アルキルジフェ ニルエーテル油 1760.0	アルキルジフェ ニルエーテル油 1300.0
増ちょう剤 (g)	有機化ベントナ イト 240.0	ポリテトラフル オロエチレン 700.0
混和ち ょう度 25℃	270	268
軸受音響 (点)	10	5
軸受潤滑寿命(h)	560	2800

As shown in embodiment, grease of the present invention comprises the longlivedness that 1200h is

older than in ASTM D 1741 bearing lubrication life test at 150 degrees Celsius both and 75 points get a high score older than in bearing sound examination by *anderonmeta*, it was superior in noise low. With comparative example 1-3, base oil aside from alkyl diphenyl ether oil was employed, but, all was short generation time. In particular, A thing using ester oil is short paddle. Because, this, affinity of ester oil and thickener is preferable, grease softens within check, what was leaked is guessed with a cause by bearing. In addition, Comparative example 4 was short generation time, too. Because, as for this (1), there are a little equation medius  $R_1$  and rate of alkyl group of carbon number 8 to occupy within  $R_3$  with 30%, and there is much rate of alkyl group of carbon number 18, it is conceivable that because of this it was leaked from grease bearing with a cause in shear stability, heat resistance.  $R_1$  in equation extends to comparative example 5,6 (1), and  $R_3$  is each aromatic, example of alicycle equation, but, although it is superior, it is inferior to high temperature, bearing lubrication life time in sound examination. Content as thickener shows less than double quantity % and result of a case of greater than 25 % by weight in each comparative example 7,8. It was less than double quantity %, and it was floating, and it was examination impotence it was filled with bearing, and to leak immediately, and it became too hard in manufacture, and it was not provided in stirring impotence or purpose grease when 25 % by weight was exceeded. In addition, It was inferior to acoustic properties with comparative example 9,10 using high-temperature type thickener aside from urea compound namely *yukika bentonaitoka* and PTFE both, and high temperature life time was not able to be achieved in application of *yukika bentonite* particularly, too. (an effect of the invention) effect grease composition of the present invention comprises longlivedness under high temperature as is apparent from the above-mentioned account because specific ampere combined diurea chemical agent of specify with the base oil which is essentially based on alkyl diphenyl ether oil and to be superior in noise characteristic low is provided.

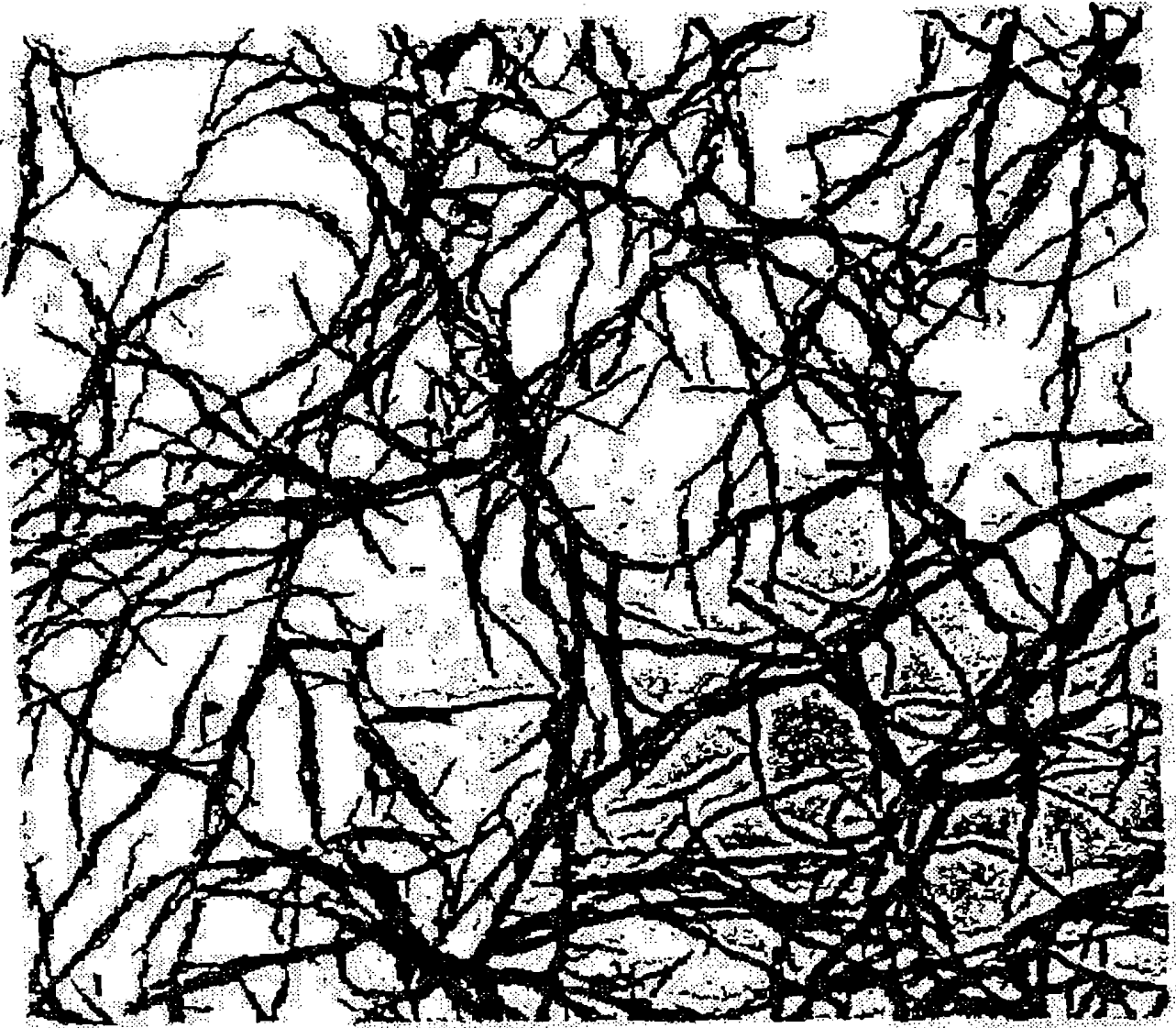
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#### [BRIEF DESCRIPTION OF DRAWINGS]

Figure 1 and figure 2 are electron microscope mirror photography of magnification 6000 times to show corpuscule configuration of thickener corpuscule of the grease which is described by grease of the present invention and Japanese Patent Application No. 63-86631 specification respectively in.

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